

CLAIMS

1. A reactor (101;301) for solid phase continuous polymerisation of polyethylene terephthalate (PET), comprising:
 - a casing (103;303);
 - 5 - a feeding line (111;311) to feed a low molecular weight PET pre-polymers flow into said reactor (101;301);
 - a gas line (121;321) to feed, through supply valves (123;323), a gas into said reactor (101;301);
 - a discharging line (141;341) inferiorly connected to the 10 bottom of the reactor (101;301) to discharge the polymerised product;
 - a circuit (131;331) connected to the reactor (101;301) to purify the gas and to recover pre-polymer particles by means of a proper separator (133;333),
- 15 characterised in that inside said reactor (101;301) means are provided to generate a plurality of fluidised stages in series (109;309) to cause an increase of the intrinsic viscosity (I.V.) of said PET pre-polymers flow.
2. A reactor according to claim 1, characterised in that said casing (103) of said reactor (101) has a substantially parallelepiped shape and it is horizontally arranged.
3. A reactor according to claim 2, characterised in that said means comprise a plurality of inferior vertical walls (105a) secured to the base (107a) of said reactor (101) and a plurality of superior vertical walls (105b) secured to the ceiling (107b) of said reactor (101), between said inferior

walls being generated said fluidised stages (109).

4. A reactor according to claim 3, characterised in that said supply valves (123) are in such a number and arranged in such a way to generate a sufficient gas flow in correspondence with 5 each fluidised stage (109).

5. A reactor according to claim 4, characterised in that said supply valves (123) associated to the same fluidised stage (109) are equipped with heating devices (125) suitable for bringing at the desired temperature the inert gas flowing 10 through the bottom (104) of the reactor (101) in correspondence with each fluidised stage (109), this way achieving a differentiated heating of said fluidised stages (109).

6. A reactor according to claim 1, characterised in that said casing (303) of said reactor (301) has a substantially 15 cylindrical shape and it is vertically arranged.

7. A reactor according to claim 6, characterised in that said means comprise a plurality of shelves (305) secured to the internal wall (307) of said casing (303) in correspondence with which said fluidised stages (309) are generated.

20 8. A reactor according to claim 7, characterised in that said shelves (305), provided inside said casing (303), are secured to said internal wall (307) of said casing (303) so to radially extend, up to about the centre line of the chamber created inside said casing (303).

25 9. A reactor according to claim 8, characterised in that said shelves (305) are preferably formed from holed metal plates.

10. A reactor according to claim 9, characterised in that said shelves (305), provided inside said casing (303), are almost horizontal.

11. A reactor according to claim 10, characterised in that 5 said shelves (305) are alternately arranged so to define an obligatory path inside said reactor (301) for the pre-polymers descending flow.

12. A reactor according to any of the preceding claims, characterised in that said fluidised stages in series (109;309) 10 are in a number of five.

13. A reactor according to claim 1, wherein said feeding line (111;311) is equipped with a device (113;313) suitable for regulating the flow-rate of fed amorphous PET pre-polymer and to prevent gas leakage.

15 14. A reactor according to claim 13, wherein said device (113;313) is a rotating volumetric distributing apparatus.

15. A reactor according to claim 1, wherein said discharging line (141;341) is equipped with a device (143;343) suitable for discharging the PET after solid phase polymerisation and to 20 prevent gas leakage.

16. A reactor according to claim 15, wherein said device (143;343) is a rotating volumetric distributing apparatus.

17. A reactor according to claim 1, wherein said circuit (131;331) further comprises a separation device (135;335) to 25 recover ethylene glycol and oligomers at the liquid state and then to recycle them upstream the overall PET manufacturing

cycle.

18. A reactor according to any of the preceding claims, characterised in that said PET pre-polymers flow has a low initial I.V. value, generally an I.V. value in the range of 5 $0.20 \div 0.45$ dl/g and preferably an I.V. value of 0.30 dl/g.

19. A reactor according to any of the preceding claims, characterised in that said I.V. increase of said PET pre-polymers flow is comprised in the range of $0.35 \div 0.65$ dl/g, preferably of 0.55 dl/g.

10 20. A reactor according to any of the preceding claims, characterised in that said I.V. increase of said PET pre-polymers flow is ≥ 0.06 dl/g.

21. A reactor according to any of the preceding claims, characterised in that said I.V. increase of said PET pre-15 polymers flow is ≥ 0.20 dl/g.

22. A reactor according to any of the preceding claims, characterised in that said PET pre-polymers flow is a PET sand flow, being the sand particle size preferably comprised in the range $60 \div 300$ μm .

20 23. A reactor according to any of the preceding claims, characterised in that said PET pre-polymers flow is a PET sand flow, being the sand particle size preferably comprised in the range $100 \div 250$ μm .

24. A reactor according to any of the preceding claims, 25 characterised in that said PET pre-polymers flow fed into the reactor (101;301) is maintained in said reactor (101;301) at a

temperature comprised in the range 200 \div 235 $^{\circ}\text{C}$, preferably of 230 $^{\circ}\text{C}$.

25. A reactor according to any of the preceding claims, characterised in that said PET pre-polymers flow fed into the 5 reactor (101;301) is maintained in said reactor (101;301) at a temperature comprised in the range 205 \div 230 $^{\circ}\text{C}$.

26. A reactor according to any of the preceding claims, characterised in that said gas is an inert gas, preferably nitrogen.

10 27. A reactor according to any of the preceding claims, wherein said gas flow inside said reactor is directed in cross-flow or in counter-current flow with respect to the flow of the PET granules that pass through said reactor.

15 28. A reactor according to any of the preceding claims, wherein the ratio between the mass of the gas flow that passes through said reactor and the mass of the PET granules in the reactor is > 0.62 .

20 29. A reactor according to any of the preceding claims, wherein the ratio between the mass of the gas flow that passes through said reactor and the mass of the PET granules in the reactor is > 0.9 .

30. A reactor according to any of the preceding claims, wherein said gas is an inert gas or air.

25 31. A reactor according to claim 30, wherein said gas is air with a dew point $< -30^{\circ}\text{C}$.

32. A reactor according to claim 30, wherein said gas is a

mixture of gases chosen from the group comprising nitrogen, noble gases, carbon dioxide, carbon monoxide and oxygen and wherein the oxygen content is < 10% by weight.

33. A reactor according to claim 30, wherein said gas is a mixture of gases chosen from the group comprising nitrogen, noble gases, carbon dioxide, carbon monoxide and oxygen and wherein the oxygen content is < 6% by weight.

34. A reactor according to any of the preceding claims, wherein the gas is recycled to the reactor, after having been purified of the organic impurities; until a level of organic impurities \leq 100 p.p.m. by weight (CH_4 equivalent) has been reached.

35. A reactor according to any of the preceding claims, wherein the PET granules have an irregular shape with a volume comprised between 0.05 and 10 mm^3 .

36. A reactor according to any of the preceding claims, wherein inside said reactor the polyester granules are subjected to a solid phase polycondensation and/or drying and/or crystallisation and/or dealdehydisation.

37. A process for solid phase continuous polymerisation of polyethylene terephthalate (PET), comprising the steps of:
- feeding a low molecular weight PET pre-polymers flow into a reactor (101;301) through a feeding line (111;311);
- feeding a gas into said reactor (101;301) through a gas line (121;321) in cross-flow or in counter-current flow with respect to said PET pre-polymers flow,

characterised in that said polymerisation is carried out in a plurality of fluidised stages in series (109;309) generated inside said reactor (101;301) to cause an increase of the intrinsic viscosity (I.V.) of said PET pre-polymers flow.

5 38. A process according to claim 37, characterised in that said polymerisation is carried out in a number of fluidised stages in series (109;309) of five.

39. A process according to claim 38, characterised in that said polymerisation is carried out at non-isothermal
10 conditions.

40. A process according to claim 38, characterised in that said polymerisation is carried out at isothermal conditions.

41. A process according to claim 38, characterised in that said polymerisation is carried out in a time period preferably
15 of about 2 hours.

42. A process according to any of the claims from 37 to 41, characterised in that said PET pre-polymers flow has a low initial I.V. value, generally an I.V. value in the range of 0.20 ÷ 0.45 dl/g and preferably an I.V. value of 0.30 dl/g.

20 43. A process according to any of the claims from 37 to 42, characterised in that said I.V. increase of said PET pre-polymers flow is comprised in the range of 0.35 ÷ 0.65 dl/g, preferably of 0.55 dl/g.

44. A process according to any of the claims from 37 to 43, 25 characterised in that said I.V. increase of said PET pre-polymers flow is ≥ 0.06 dl/g.

45. A process according to any of the claims from 37 to 44, characterised in that said I.V. increase of said PET pre-polymers flow is ≥ 0.20 dl/g.

46. A process according to any of the claims from 37 to 45, 5 characterised in that said PET pre-polymers flow is a PET sand flow, being the sand particle size preferably comprised in the range 60 \div 300 μm .

47. A process according to any of the claims from 37 to 46, characterised in that said PET pre-polymers flow is a PET sand 10 flow, being the sand particle size preferably comprised in the range 100 \div 250 μm .

48. A process according to any of the claims from 37 to 47, characterised in that said PET pre-polymers flow fed into the reactor (101;301) is maintained in said reactor (101;301) at a 15 temperature comprised in the range 200 \div 235 $^{\circ}\text{C}$, preferably at 230 $^{\circ}\text{C}$.

49. A process according to any of the claims from 37 to 48, characterised in that said PET pre-polymers flow fed into the reactor (101;301) is maintained in said reactor (101;301) at a 20 temperature comprised in the range 205 \div 230 $^{\circ}\text{C}$.

50. A process according to any of the claims from 37 to 49, characterised in that said gas is an inert gas, preferably nitrogen.

51. A process according to any of the claims from 37 to 50, 25 wherein said gas flow inside said reactor is directed in cross-flow or in counter-current flow with respect to the flow of

said PET granules that pass through said reactor.

52. A process according to any of the claims from 37 to 51, wherein the ratio between the mass of the gas flow that passes through the reactor and the mass of the PET granules in the 5 reactor is > 0.62 .

53. A process according to any of the claims from 37 to 52, wherein the ratio between the mass of the gas flow that passes through the reactor and the mass of the PET granules in the reactor is > 0.9 .

10 54. A process according to any of the claims from 37 to 53, wherein said gas is an inert gas or air.

55. A process according to claim 54, wherein said gas is air with a dew point $< -30^{\circ}\text{C}$.

15 56. A process according to claim 54, wherein said gas is a mixture of gases chosen from the group comprising nitrogen, noble gases, carbon dioxide, carbon monoxide and oxygen and wherein the oxygen content is $< 10\%$ by weight.

20 57. A process according to claim 54, wherein said gas is a mixture of gases chosen from the group comprising nitrogen, noble gases, carbon dioxide, carbon monoxide and oxygen and wherein the oxygen content is $< 6\%$ by weight.

58. A process according to any of the claims from 37 to 57, wherein the gas is recycled to the reactor, after having been purified of the organic impurities, until a level of organic 25 impurities ≤ 100 p.p.m. by weight (CH₄ equivalent) has been reached.

59. A process according to any of the claims from 37 to 58, wherein the PET granules have an irregular shape with a volume comprised between 0.05 and 10 mm³.

60. A process according to any of the claims from 37 to 59, wherein inside said reactor the polyester granules are subjected to a solid phase polycondensation and/or drying and/or crystallisation and/or dealdehydisation.

61. A process according to any of the claims from 37 to 60, characterised in that ethylene glycol and oligomers present at 10 the end of said polymerisation are recovered at the liquid state in a separation device (135;335) provided in a circuit (131;331) connected to the reactor (101;301) and then recycled upstream the overall PET manufacturing cycle.

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